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(71) Applicants *(for all designated States except US)*: SMITHKLINE BEECHAM CORPORATION [US/US]; One Franklin Plaza, Philadelphia, PA 19103 (US). SMITHKLINE BEECHAM PLC [GB/GB]; New Horizons Court, Great West Road, Brentford, Middlesex TW8 9EP (GB).

(72) Inventors; and

(75) Inventors/Applicants *(for US only)*: CASH, Michael [US/US]; 238 Mechanic Street, Boonton, NJ 07005 (US). DAVIS, Gregory [GB/GB]; 24 Waltham Road, Twyford, Berkshire RD10 9EF (GB). DESAI, Indrajit [US/US]; 22 Clearfield Road, Succasunna, NJ 07876 (US). FORWARD, Geoffrey, C. [GB/GB]; The Junipers, Headly Road, Epsom Downs, Surrey KT18 6BW (GB). LAYER, Teresa [GB/GB]; 7 Fountain Gardens, Osborne Road, Windsor, Berkshire SL4 3SZ (GB). MCCONVILLE, Peter, S. [GB/GB]; 74 Amhurst Gardens, Isleworth, Middlesex TW7 6AJ (GB).

(74) Agents: STEIN–FERNANDEZ, Nora et al.; SmithKline Beecham Corporation, Corporate Intellectual Property, UW2220, 709 Swedeland Road, P.O. Box 1539, King of Prussia, PA 19406–0939 (US).

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(54) Title: TOOTH WHITENING PREPARATIONS

(57) Abstract

This invention relates to compositions for preventing the build–up of stain and whitening teeth and dental prostheses using a water soluble alkali metal tripolyphosphate in combination with an alkali metal pyrophosphate salt, and optionally polyvinyl pyrrolidone.

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Tooth Whitening Preparations

Field of the Invention

This invention relates to an oral hygiene composition and methods for whitening of and preventing staining on natural teeth and dental prostheses. The whitening is achieved through the use of an alkali metal tripolyphosphate at about between 0.5 to 10 percent by weight in combination with at least one alkali metal pyrophosphate salt at about 0.1 to 10 percent by weight. Whitening and stain-prevention are achieved through the use of an alkali metal tripolyphosphate at about between 0.5 to 10 percent by weight in combination with at least one alkali metal pyrophosphate salt at about 0.1 to 10 percent by weight, and polyvinyl pyrrolidone ("PVP") at about 0.1 to 10 percent by weight in a dentally acceptable carrier. Any orally acceptable presentation, or one suitable for dental prostheses, can be utilized in this invention.

Background of the Invention

Several factors contribute to enamel discoloration, but the three main factors are believed to be: (i) formation of plaque and tartar matrices on the tooth surface which then entraps stains or stain attachment to natural pellicle; (ii) ingestion of certain drugs during gestational tooth formation; and (iii) discoloration due to oral cavity traumatization following which blood break-down products seep into the mineralized area of the teeth during enamel formation. This invention is primarily concerned with the first factor of tooth discoloration, that is, the natural stain which accumulates on teeth.

Over-the-counter teeth whitening preparations have been developed to address the cosmetic preference of many to restore luster to tooth enamel discolored by surface entrapped materials. While all dentifrices and mouthwashes contain some cleaning and polishing agents, some enamel deposits become intractable to being fully removed by these agents under normal use conditions. Also, these preparations may not be formulated with the amount or type of agent required to fully remove the amount of stains and discoloration which build up due to excessive exposure to various staining agents. For example, smokers often develop discolored enamel because the tars and particulate in exhaled cigarette smoke collect on the teeth. Further, a number of comestibles, such as tea, or some medicinal agents, can stain or discolor tooth enamel.

There are various approaches to enamel whitening currently in general use. One approach is a physical abrading of the stain to effect stain removal. Harsher abrasives, also known as polishing agents, than those used in typical non-whitening

toothpaste preparations, are employed in this approach. Most, if not all of these preparations are toothpastes, gels or powder dentifrices. Brushing or similar scrubbing or polishing action is required as a complement to successful stain removal. Examples of such products are Smokers Topol made by Topol-Dep Corporation and marketed to smokers and tea drinkers as a means for removing stains caused thereby.

Oxidizing agents represent one of the most widely distributed and utilized agent in oral preparations; all of these products being pastes or gels. Urea peroxide, hydrogen peroxide or calcium peroxide are the most commonly used oxidizing agents in these products. These treatments require some time to achieve good results; typically one and one-half to eight days, or two to three months, depending on the peroxide source and its concentration.

Recently catalytic systems have come back into favor and have been packaged and marketed through retail outlets in parallel with other oral care products. Proteolytic enzymes are the catalyst of choice, particularly papain. A second active such as a citric acid salt has been used by at least one manufacturer. These products are presented in a paste or gel formulation. The products are claimed to whiten teeth by removing the plaque which has entrapped the stain.

European Patent Application EP 248936A, published December 16, 1987, discloses a cleaning tablet for dentures comprising two layers, each with a different composition, characterized by a change in the pH from acidic to alkaline during the dissolving process of the tablet in water. The first layer contains 25-35% $NaHCO_3$ as the carbon dioxide producing agent, and, *inter alia*, 3-15% disodium pyrophosphate. The second layer contains 25-45% sodium perborate monohydrate, 25-35% potassium monopersulphate as an oxidzing agent, and *inter alia*, 7-18% sodium tripolyphosphate. The advantage asserted is that the tablet shows a characteristic pH swing from acidic to alkaline and a greater generation of oxygen than commercially available two-phase tablets.

Japanese Application No. 57-111996, published January 6, 1984, discloses a dentifrice composition containing pyrogenic silica (silicas made by heating), to which is added 0.01-1 weight percent of one or more phosphoric acid salts selected from orthophosphates, polyphosphates and metaphosphates, particularly from potassium pyrophosphate, sodium pyrophosphate, trisodium pyrophosphate, disodium pyrophosphate, sodium polyphosphate, sodium tripolyphosphate, potassium tripolyphosphate, potassium metaphosphate, sodium trimetaphosphate, sodium hexametaphosphate and sodium heptametaphosphate. The advantage of this

particular formulation is alleged to be that the astringent taste of the pyrogenic silica is effectively suppressed, providing a dentifrice with a good mouth feel.

U.S. Patent 4,913,895, issued April 3, 1990, to Lion Corp., discloses an oral hygiene composition alleged to have a synergistic antibacterial effect against Actinomyces viscosus, a plaque-forming bacteria causing calculus formation and periodontal disease. The composition contains 0.1 to 10% by weight of component (1) which is a polyphosphate selected from the group consisting of sodium pyrophosphate, potassium pyrophosphate, sodium tripolyphosphate, potassium tripolyphosphate, sodium tetrapolyphosphate, potassium tetrapolyphosphate, sodium metaphosphate, potassium metaphosphate, sodium trimetaphosphate, potassium trimetaphosphate, sodium hexametaphosphate, potassium hexametaphosphate, and mixtures, the synergistically effective improvement consisting of the step of including with said anticalculus polyphosphate 0.01 to 2% by weight of component (2) which is a member selected from the group consisting of 1-menthol, anethol, and mixtures thereof.

U.S. Patent 4,923,684, issued May 8, 1990, to Ibrahim et al., describes a storage stable anticalculus toothpaste composition comprising at least about 4% by weight of STP.

PCT/US94/09185, published February 23, 1995, relates to a composition for whitening teeth and dentures containing sodium tripolyphosphate and a reducing agent such as Vitamin C.

PCT/US94/14662, published June 29, 1995, relates to a composition for whitening teeth and dentures containing STP alone; STP combined with peroxides; STP combined with enzymes (papain); and STP combined with peroxides and enzymes.

U.S. Patent 5,538,714 issued July 23, 1996, to Pink et al., describes a method of reducing the adherence of oral bacteria to tooth enamel comprising applying to the tooth enamel a composition containing an anti-adherence effective amount of PVP.

GB 739,936, published November 2, 1955, discloses compositions containing both chlorophyll and water-soluble polyvinyl pyrrolidone for inhibiting or preventing the formation of greenish stains associated with chlorophyll on certain absorbant materials such as cellulose, animal and synthetic fibers.

GB 741,315, published November 30, 1955, discloses dentifrices containing polyvinyl pyrrolidone as a stain remover, particularly for tar-like stains.

While tetrapotassium and tetrasodium pyrophosphate are known as anticalculus agents, their use in combination with sodium tripolyphosphate and

optionally PVP, to either whiten teeth or dental prostheses or to prevent the stain build-up on teeth and dental prostheses, has not been disclosed before. Calculus is a deposit, comprising an organic and an inorganic portion, forming on the surfaces of teeth at the gingival margin. The inorganic portion consists mainly of calcium phosphate arranged in a hydroxy-apatite crystal lattice structure, whereas the organic portion consists of desquamated epithelial cells, leukocytes, food debris and various types of microorganisms. As the calculus develops it becomes visibly white or yellowish in color or it may become stained by some extraneous agency.

This invention provides a unique alternative to tooth whitening and stain prevention. It utilizes an alkali metal tripolyphosphate salt in combination with at least one alkali metal pyrophosphate salt, and PVP, to effect stain removal, whiten tooth enamel, and prevent future staining of the teeth. If tooth whitening or the removal of stains from teeth are desired alone, without the prevention of stain build-up, a useful composition of the invention is an alkali metal tripolyphosphate salt in combination with at least one alkali metal pyrophosphate salt.

Surprisingly, according to soaking studies, the use of one or more of an alkali metal pyrophosphate in combination with sodium tripolyphosphate showed significant increases in whitening effect over the whitening effect of sodium tripolyphosphate when used alone as a whitening agent. In addition, and suprisingly, according to soaking studies, the use of one or more of an alkali metal pyrophosphate in combination with sodium tripolyphosphate and PVP, showed significant increases in stain prevention and whitening effect over the whitening effect of sodium tripolyphosphate when used alone as a whitening agent. Furthermore, it is noted that, by using such a combination of an alkali metal pyrophosphate salt with a tripolyphosphate salt and optionally PVP, lower concentrations of the tripolyphosphate salt can be used, while enhancing the whitening effect of the tripolyphosphate salt when used alone. When PVP is used in combination with sodium tripolyphosphate and one or more of an alkali metal pyrophosphate salt, one achieves the additional effect of reducing the build-up of stain as well as the whitening effect.

Summary of the Invention

This invention comprises compositions for preventing, reducing or removing surface deposited stains from natural teeth and dental prostheses comprising a dentally acceptable preparation comprising about 1 to 10% by weight of a water soluble alkali metal tripolyphosphate in combination with about 0.5 to 10% by weight of at least one alkali metal pyrophosphate salt and about 0.1 to 10% by weight of PVP.

In addition this invention relates to a method for preventing, reducing or removing surface deposited stains from natural teeth and dental prostheses which method comprises contacting teeth or dental prostheses with a whitening effective amount of the instant dentally acceptable composition, in particular, a composition comprising at least about 1 to 10% by weight of a water soluble alkali metal tripolyphosphate in combination with about 0.5 to 10% by weight of at least one alkali metal pyrophosphate salt and about 0.1 to 10% by weight of PVP.

This invention also comprises compositions and a method for whitening or removing surface deposited stains from natural teeth and dental prostheses comprising contacting the teeth or dental prostheses with a dentally acceptable preparation comprising about 1 to 10% by weight of a water soluble alkali metal tripolyphosphate in combination with about 0.5 to 10% by weight of at least one alkali metal pyrophosphate salt.

Brief Description of the Figures

Fig. 1 and Fig. 1A: depict graphically a comparison of the increase in L value obtained with varying concentrations of a combination of disodium pyrophosphate and sodium tripolyphosphate versus sodium tripolyphosphate alone.

Fig. 2 and Fig. 2A: depict graphically a comparison of the increase in L value obtained with varying concentrations of a combination of tetrapotassium pyrophosphate and sodium tripolyphosphate versus sodium tripolyphosphate alone.

Fig. 3: depicts graphically an increase in L value obtained with a combination of sodium tripolyphosphate and disodium pyrophosphate compared with the increase in L value of sodium tripolyphosphate alone and disodium pyrophosphate alone.

Figs. 4-8: depict, in chart format, the data used to prepare Figs. 1, 1A, 2 and 2A.

Fig. 9: depicts data obtained from a stain prevention study to determine the stain-preventing properties of various toothpastes and solutions.

Fig. 10: depicts data from a soaking study to examine whitening effects of various formulations.

Detailed Description of the Invention

It has now been discovered that certain alkali metal pyrophosphate salts, in particular, tetrasodium pyrophosphate and tetrapotassium pyrophosphate each in combination with sodium tripolyphosphate ("STP"), are highly effective whitening agents. In addition, it has been discovered that PVP is a highly effective stain

prevention agent, particularly when used in a toothpaste matrix which gives suprisingly better stain prevention results than when compared with PVP in solution.

Specifically disclosed is an oral composition, purported to be effective in inhibiting or retarding the formation of dental calculus without adversely affecting tooth structure, the composition comprises from 0.5 to 10% by weight of an alkali metal pyrophosphate salt in combination with from about 1 to 10% by weight of an alkali metal tripolyphosphate and a carrier suitable for use in the oral cavity, the pH being in the range from 6.5 to 10.

These following words are intended to be given the same meaning here as would be accorded to them in their contemporary usage in the oral and dental care arts. More specific usage for the invention herein is described below.

The terms "stain" or "staining" are used interchangeably with discoloration and generally mean that the surface of the enamel (or prostheses) has taken on some unwanted or unnatural coloration distinct from the color of the underlying enamel.

The phrases "stain removal" is used herein to mean removing stain which is periodically deposited on the tooth surface. Stain removal is effectuated by the whitening agents of this invention, i.e., STP and the alkali metal pyrophosphates, which in combination give enhanced whitening results when compared with STP alone.

The phrase "stain prevention" is used herein to mean inhibit or reduce the build-up of stain on the tooth surface.

Without being limited to a particular mechanism of action, we propose the following theory of stain prevention for this invention. PVP is a known complexing/sequestering agent for tannins. Tannins from various food sources causes staining of teeth. PVP, in a toothpaste matrix with a detergent such as sodiumlaurylsulfate (SLS), forms a film on the tooth surface and is thus retained on the tooth. Therefore, by using the inventive composition, and contacting the tooth surface therewith, stains introduced into the oral cavity might actually be sequestered by the PVP remaining as a film on the tooth surface, and not adhere to the tooth surface. As the PVP is sloughed-off the tooth surface, the sequestered stain is taken with it. It is the object of the present invention to provide a composition containing a combination of an alkali metal pyrophosphate salt, an alkali metal tripolyphosphate and PVP, which has a surprising capacity to effect both stain removal and stain prevention, and thus whiten tooth enamel.

One active component of this invention comprises a water soluble alkali metal tripolyphosphate. The sodium form of this salt is preferred, although the potassium or mixed sodium and potassium salts could be used as an embodiment as

well. All physical forms of the salt can be used, e.g., a hydrate or the dehydrated form. STP is available from Monsanto Corp. or Sigma Chemical.

The amount of tripolyphosphate salt will be between about 0.5 and 10% by weight of the preparation. A preferred amount of said salt is between about 5% and 7.5% by weight.

Accordingly, in a first aspect of the invention there is provided a composition for use in whitening human teeth and dental prostheses comprising tetrasodium pyrophosphate, tetrapotassium pyrophosphate, STP and PVP. In a second aspect of the invention there is provided a composition for use in preventing stain build-up on human teeth and dental prostheses comprising tetrasodium pyrophosphate, tetrapotassium pyrophosphate, STP and PVP. In a third aspect of the invention there is provided a composition for use in whitening human teeth and dental prostheses comprising at least one alkali metal pyrophosphate and STP.

The disodium pyrophosphate salt (available from Monsanto Corp. or Sigma Chemical, and also known as disodium dihydrogen pyrophosphate) is incorporated into the formulation in the range between about 0.1 and 10% by weight of the preparation, suitably between about 0.75 and 10% by weight, preferably between about 2.0 and 6.0% by weight, and most preferably about 1.0 to 2.0% by weight. Disodium pyrophosphate in combination with STP is a preferred whitening composition. A particularly preferred whitening composition of the invention comprises about 1.0 to 2.0% disodium pyrophosphate and 5 to 7.5% STP.

The tetrasodium pyrophosphate salt (available from Monsanto Corp. or Sigma Chemical) is incorporated into the formulation in the range between about 0.1 and 10% by weight of the preparation, suitably between about 0.75 and 10% by weight, preferably between about 0.75 and 4.0% by weight, and most preferably about 0.75 to 2.0% by weight.

The tetrapotassium pyrophosphate salt (available from FMC Corporation, and also known as tetrapotassium salt/diphosphoric acid) is incorporated into the formulation in the range between about 0.1 and 10% by weight of the preparation, preferably 1.0 and 10% by weight, more preferably between about 1.0 and 4.0% by weight, and most preferably between 1.0 and 3.0% by weight. A particularly preferred whitening composition of the invention comprises about 3.0 to 4.0% tetrapotassium pyrophosphate and 5 to 7.5% STP.

PVP suitable for use in the present invention preferably has an average molecular weight in the range 5,000 to 100,000. A suitable grade of PVP with an average molecular weight of 50,000 is available commercially from BASF Corporation and is known as Povidone K30. PVP is incorporated into the

formulation in the range between about 0.1 to 10% by weight, preferably between about 0.5 to 5.0% by weight and most preferably between about 0.5 to 2.0% by weight.

The alkali metal pyrophosphate salt portion of the instant composition can be either a single pyrophosphate salt, or a combination of pyrophosphate salts. A particularly preferred composition of the invention comprises about 0.75 to 2.0% tetrasodium pyrophosphate, 1.0 to 3.0% tetrapotassium pyrophosphate, 5 to 7.5% STP and 0.5 to 2.0% PVP. The most preferred embodiment of this invention comprises about 2% tetrapotassium pyrophosphate, 0.91 % tetrasodium pyrophosphate, 5% STP and 1% PVP.

The whitening and stain preventing compositions may be presented in any of the conventional formulations such as a dentifrice, including a toothpaste, a mouthwash or a formulation that is chewed or sucked by the user such as a lozenge or a chewing gum.

These formulations will be presented so that they are safe for use in the oral cavity and will not have a deleterious effect if accidentally swallowed. The oral care art has developed a substantial body of formulation types and has identified and tested a large list of ingredients useful in these formulations. Confecting or manufacturing these preparations, and their safe packaging and storage is also well documented.

For achieving optimum whitening and stain preventing results the formulation as used should have a pH of between about 7.0 to 9.0. Optimum cleaning results are achieved with a pH of about 8 with reference to how the formulation is used for brushing teeth in the mouth. For example, a toothpaste preparation will usually be diluted in the mouth by about 1 plus 2 or 1 plus 3 volumes of water/saliva while brushing. Thus a paste or gel, for example, optimized for whitening will be formulated to achieve a pH between about 7 and 9 when it is being actually used to brush or treat teeth. An acid or base may be used to adjust the pH of the preparation; the choice is within the skill of the art concerned with oral preparations. The optimum pH for stability purposes in a particular preparation, prior to use, may vary from this range and may have a different optimum pH, depending on excipients and additives, all of which is within the skill of the art.

In addition to the active ingredients, formulations for toothpastes, liquid pastes, gels and toothpowders suitable for this invention will contain the usual carriers, binders, surfactants, humectants, coloring agents, pigments, antiplaque agents, anti-bacterial agents, bioadhesive-type agents, abrasives, anticaries agents, flavorings, sweeteners, bulking agents, and the like.

Suitable abrasives for use in the present invention include precipitated silica, plastics particles, alumina, calcium carbonate, and zinc orthophosphate, insoluble metaphosphates and calcium pyrophosphate. Pyrogenic silicas are not claimed as a useful silica for the instant invention. Silica is an especially preferred abrasive for use herein.

The patent and scientific literature is replete with examples of such abrasives. U.S. Patent 4,822,599 listing a series of dentifrice abrasives, also references commercial sources and methods for their preparation.

Silica abrasives are well known and commercially available, generally having an average particle size ranging between about 0.1 to about 30 microns, such as from about 5 to about 15 microns. Silica dental abrasives useful in the present invention include those marketed by the J.M. Huber Corporation under the trade name Zeofree™ (Zeodent 113) and the silica xerogels marketed by the W.R. Grace and Company, Davison Chemical Division under the trade name 'Syloid'. U.S. Patent 3,358,230 and U.S. Patent 3,862,307 describe silica dental abrasives useful in the toothpaste compositions according to the present invention. The silica abrasive may also be a naturally occurring amorphous silica such as diatomaceous earth. Suitable forms of diatomaceous earth are those marketed under the trade mark 'Celite' by Johns-Manville Products Corporation, for instance 'Celite Superfine Superfloss'.

The selected abrasive should be compatible with the phosphate actives, as well as any additives which may be actives as well, such as fluoride ions and antibacterial agents. In addition, as with any other paste, gel or powder, the selection of an abrasive can be influenced by the consequence of combining a particular abrasive with another additive. For example, if fluoride ions and calcium pyrophosphate ions are to be included in these preparations the pyrophosphate should be converted from its γ-phase to its β-phase by heating the γ-phase to 700°-900°C as per the teachings of U.S. Patent 3,112,247. Also certain quaternary ammonium-based antibacterial agents may not be compatible with some silica abrasives.

Plastics dental abrasives are well known and are described in, for example, GB 939 230, GB 995 351, GB 1 055 784, and U.S. Patent 3,151,027.

Alumina abrasives are well known and commercially available. Preferably the alumina abrasive may be treated with a solution of a surface-treating agent which may be an alkali metal silicate, hydrogen peroxide, an acid or an organophosphorus compound, of which an alkali metal silicate is especially preferred, as described in U.S. Patent 4,781,982 (to Aluminum Company of America).

A calcium carbonate abrasive is preferably used in conjunction with an ionic agent to suppress the formation of free calcium ions, such as an alkali metal carbonate or bicarbonate, or mixture thereof, as described in EP 0 092 929 (to Beecham Group p.l.c.).

Abrasive concentrations can cover a very broad range. Preparations are described with abrasive ranging in concentration from 5 to 80% by weight depending on the abrasive. A secondary concentration range is that of 10 to 50% depending on the abrasive selected. Herein the preferred abrasive, silica, is employed in amounts between 5 and 30% by weight.

A source of fluoride ion may be included in the instant composition. Fluoride ion sources are numerous. For example, see U.S. Patent 3,535,421 listing numerous salts useful in the dental arts. While any one of these sources could be used, sodium fluoride, stannous fluoride and sodium monofluorophosphate are considered the preferred ion sources in most dentifrices.

Fluoride ions are routinely added into dentifrices in an amount sufficient to provide up to 3500 ppm, preferably 1100 ppm of the fluoride ion. Where a preparation is formulated such that the fluoride ion is confined to one component of the preparation, but is mixed with the other components at the time of use, the fluoride ion source should be adjusted upward in an amount sufficient to provide a concentration of up to about 3500 ppm, but preferably 1100 ppm, in the product as used.

Suitably, in compositions of the present invention, the orally acceptable vehicle may comprise other components such as, flavorings, coloring agents, sweeteners, humectants, thickening agents, binders and surfactants.

Binders and thickening agents can be added to assure physical integrity in pastes, gels and liquid pastes. Preferred thickening and binding agents include for example natural and synthetic gums such as xanthan and acacia gums, carageenans, alginates, cellulose ethers and esters such as carboxy methyl cellulose, polyoxyalkyl polymers such as the Pluronics polymers, certain polymers exemplified by the carboxyvinyl polymers (Gantrez and the like), and silica. When the abrasive is silica, it is preferred to use a thickening silica as the thickening agent. A preferred thickening silica for use herein is Zeofree™ 153, which is a precipitated synthetic amorphous silica.

Binders are usually added in amounts ranging between 0.1 and 5.5% by weight.

Humectants are added to gels and pastes to prevent their drying out on exposure to air. In addition, they impart a "moist" feel to the mouth when brushing.

Some humectants, e.g., sorbitol, are perceived as sweet. Examples of compounds useful as humectants in dentifrices are the polyhydric alcohols such as glycerin, sorbitol, propylene glycol and polyethylene glycols. A preferred humectant system consists of glycerin, sorbitol (usually 70% sorbitol/water) and polyethylene glycol, which is present in an amount of about 25-45%, preferably 37-40% of the total composition. In pastes and gels one to three humectants are usually used in amounts between about 10 and 80%. Preferably the humectants are used in amounts between about 20 and 50% of the total composition.

In addition, the orally acceptable vehicle may optionally comprise surfactants, sweetening agents, flavoring agents, anticaries agents (in addition to a fluoride ion source provided as a phosphatase enzyme inhibitor), anti-plaque agents, anti-bacterial agents such as triclosan or cetyl pyridinium chloride, tooth desensitizing agents, coloring agents and pigments.

Surfactants normally are added to dentifrices to assist with removing debris. Useful surfactants include the water-soluble salts of alkyl sulfates having from 10 to 18 carbon atoms in the alkyl moiety, such as sodium lauryl sulfate, but other anionic surfactants may also be used, e.g., non-ionic, zwitter-ionic, cationic and amphoteric surfactants. These compounds, and those which are most useful in the dental arts, are well documented in the literature. Reference is made to U.S. Patent 4,822,599 for a detailed listing of useful surfactants. Surfactants are available through a number of commercial manufacturers or can be made by well documented processes.

Surfactants are normally used in amounts between about 0.5 and 5% by weight in pastes and gels but may be used at higher concentrations in some dental powders. Surfactants can also be used as gelling agents.

Taste is provided by adding a small amount of a flavoring agent to the composition. Numerous minty flavored agents are available for use in dentifrices. It is well known in the art how to select a flavoring agent which will be acceptable to consumers. Flavoring agents are routinely used at levels of between about 0.1 to 5% by weight.

Dyes, lakes and titanium dioxide are routinely used in the dentifrice arts for imparting color to the composition. When titanium dioxide is the coloring agent, a white paste or powder is obtained. These materials are widely available and are well known to the dental artisan. Coloring agents are usually present in concentrations ranging between 0.0001 and 5%.

Sweeteners are routinely added to increase consumer acceptability. Artificial sweeteners are used today to avoid the cariogenic potential of most sugars and other sweetening agents. Examples of non-cariogenic sweeteners now in routine use are

saccharin, aspartame, *D*-tryptophan, dihydrochalcones, cyclamates, xylitol and acesulfame. Sweeteners comprise about 0.1 to 5% by weight of the formulation.

Appliqués can provide an effective means for removing stains as per this invention. These can be prepared in the form of a doughy or tacky material which can be readily molded to conform to the teeth. It can then be manually compressed on the teeth as is or placed in a plastic retainer, inserted into the mouth and bitten into, and left in place of a some time, for example 15 to 30 minutes. When the appliqué is removed the debris causing the stain will be removed. The appliqué is then discarded.

The active(s) can be formulated as a mouthwash or mouth rinse as well. A mouth wash or rinse will contain up to 95% water, up to 30 % alcohol, flavor, polyhydric alcohols, anti-caries agents, plaque removing agents, sweeteners, dyes and lakes, and a preservative in some instances, and sufficient water to make volume. The active could also be incorporated into currently existing formulations such as Cepacol (Lakeside Pharmaceuticals), Plax, (Pfizer), Scope (Procter & Gamble), and the like.

A soaking and cleaning solution for dental pieces can also be prepared with the combination of active ingredients. It is contemplated that such preparations would contain water, a surfactant, an effervescing agent, and other optional ingredients. Dental prostheses would be removed and placed in a solution containing the tripolyphosphate salt and pyrophosphate salt, and soaked for several hours, then either brushed with a recommended dentifrice or simply rinsed and reinserted into the mouth.

When the preferred aqueous orally acceptable dental vehicle is employed, a toothpaste composition of the present invention suitably contains from about 10 to about 80% humectant, from about 0.25 to about 5% detergent, from 0 to about 5% sweeteners and flavoring agents together with water and an effective amount of binding and thickening agents, such as from about 0.1% to about 12%, to provide the toothpaste of the invention with the desired stability and flow characteristics.

Conventional manufacturing techniques are employed to prepare a whitening toothpaste with the inventive active combination. Toothpaste compositions of the present invention may also be prepared in the form of a clear gel or a paste of a uniform color or in the form of a striped toothpaste. A suitable apparatus for filling toothpaste tubes with striped toothpaste is described in GB 962 757. In accordance with this patent, toothpastes of different colors are fed through separate tubes of a bundle of tubes that is inserted into a toothpaste container and gradually moved relative to the container as the container is filled.

The toothpaste of the invention is used in a conventional manner by applying the toothpaste to the teeth. Most dentists and researchers recommend brushing one's teeth for at least three minutes per brushing to achieve maximum results, although compliance with this standard is not universal. A similar standard is recommended for the instant pastes and gels, although it is expected that non-compliance will still provide the desired results with regular use, i.e., daily use.

The following examples are provided by way of illustration and are not intended to limit the scope of the invention.

Example 1

Formulation of Tooth Whitening/Stain-Preventing Toothpaste

A whitening and stain-preventing toothpaste representative of what may be prepared for the practice of this invention was prepared as per the ingredient profile and percentages in Tables I and II.

Table I

(_5% STP)

INGREDIENT	CONCENTRATION
Sodium Tripolyphosphate (STP)	5.00
Tetrasodium pyrophosphate	1.00
Tetrapotassium Pyrophosphate	2.00
PVP	1.00
Sorbitol, 70%	26.00
Abrasive silica	14.00
Glycerine	10.00
Thickening silica	6.00
Polyethylene glycol 400, NF	3.00
Sodium Lauryl Sulfate	1.15
Titanium Dioxide/Dyes	1.50
Sodium Saccharin	0.20
Xanthan Gum	1.00
Flavors	1.00
Sodium Fluoride	0.24
Sodium Hydroxide	0.50
Water, DI	to 100

Table II
(7.5% STP)

INGREDIENT	CONCENTRATION
Sodium Tripolyphosphate (STP)	7.50
Tetrasodium pyrophosphate	1.00
Tetrapotassium Pyrophosphate	2.00
PVP	1.00
Sorbitol, 70%	26.00
Abrasive silica	14.00
Glycerine	10.00
Thickening silica	6.00
Polyethylene glycol 400, NF	3.00
Sodium Lauryl Sulfate	1.15
Titanium Dioxide/Dyes	1.50
Sodium Saccharin	0.20
Xanthan Gum	1.00
Flavors	1.00
Sodium Fluoride	0.24
Sodium hydroxide	0.45
Water, DI	to 100

Example 2

Formulation of Tooth Whitening Toothpaste

A whitening toothpaste representative of what may be prepared for the practice of this invention was prepared as per the ingredient profile and percentages in Tables III and IV.

Table III
(5% STP)

INGREDIENT	CONCENTRATION
Water, DI	25.91
Sorbitol, 70%	26.00
Abrasive silica	12.00
Sodium Tripolyphosphate (STP)	5.00
Disodium Pyrophosphate	5.00

INGREDIENT	CONCENTRATION
Glycerine	10.00
Thickening silica	7.5
Polyethylene glycol 400, NF	3.00
Sodium Lauryl Sulfate	1.15
Titanium Dioxide/Dyes	1.50
Sodium Saccharin	0.2
Xanthan Gum	1.0
Flavors	1.0
Sodium Fluoride	0.24
Sodium Hydroxide	0.50
Total	100.00

Table IV
(7.5% STP)

INGREDIENT	CONCENTRATION
Water, DI	23.46
Sorbitol, 70%	26.00
Abrasive silica	12.00
Sodium Tripolyphosphate (STP)	7.5
Tetrapotassium Pyrophosphate	5.0
Glycerine	10.00
Thickening silica	7.5
Polyethylene glycol 400, NF	3.00
Sodium Lauryl Sulfate	1.15
Titanium Dioxide/Dyes	1.50
Sodium Saccharin	0.2
Xanthan Gum	1.0
Flavors	1.0
Sodium Fluoride	0.24
Sodium Benzoate	
Sodium hydroxide	0.45
Total	100.00

Examples 3 & 4

Formulation of Mouthwash

A whitening mouthwash representative of what may be prepared for the practice of this invention was prepared as per the ingredient profile and percentages in Tables III and IV.

INGREDIENT	CONCENTRATION	CONCENTRATION
Water, DI	89.00	87.55
Ethanol 96%	8.00	8.00
Cetyl pyridinium chloride	0.05	0.05
Sodium fluoride	0.05	0.00
Sodium saccharin	0.05	0.05
Hydrogentated castor oil	0.2	0.2
Flavor	0.15	0.15
STP	1.25	2.5
Tetrapotassium pyrophosphate	0.50	1.00
Tetrasodium pyrophosphate	0.25	0.50
PVP	0.50	0.00
Total	100.00	100.00

Example 5 & 6

Formulation of Whitening Gum

A whitening gum representative of what may be prepared for the practice of this invention was prepared as per the ingredient profile and percentages in Tables III and IV.

INGREDIENT	CONCENTRATION	CONCENTRATION
Chewing gum base	30.00	30.00
Xylitol	15.00	15.00
Sorbitol powder	27.5	32.00
Sorbitol 70% solu.	16.00	16.00
Glycerin	2.00	2.00
Flavor	1.50	1.50
STP	4.00	2.00
Tetrapotassium pyrophosphate	2.00	1.00
Tetrasodium pyrophosphate	1.00	0.50

INGREDIENT	CONCENTRATION	CONCENTRATION
PVP	1.00	0.00
Total	100.00	100.00

Tooth whitening was measured by the following soaking experiment procedure:

A: EXTRACTION OF BOVINE TEETH

Bovine teeth were sourced from abattoirs. They were cut from jaws and sorted and selected to have initial L-values between 50 and 75 by comparison with standards provided. The teeth were immediately placed in thymol /water (1 to 100) and after shipment stored in a fridge.

The teeth were de-pulped with the help of a stainless steel barb, made by the Beutelrock Company, and purchased through Victor Dental of Clifton, NJ. To de-pulp teeth, a barb was inserted into the base of the tooth, twisted, and the pulp pulled out when the barb was removed. (Note: it may require several tries for complete removal).

The de-pulped teeth were placed back into the thymol solution, and allowed to soak for at least one hour to disinfect, after which time, the teeth were rinsed with deionized water and allowed to air dry for approximately 20 minutes.

B: MOUNTING OF BOVINE TEETH

To mount teeth the following materials were used: Jet acrylic powder, and Jet acrylic liquid, made by Lang Dental Manufacturing; plastic 100 mL beakers; bottle (mounting) caps, each 3.5 cm in diameter by 1.5 cm deep; and a stainless steel spatula. The acrylic was stored in the freezer along with plastic beakers.

A beaker was taken out of the freezer, into which 9.0 grams of acrylic powder was weighed. To the powder was added 3.1 mL of acrylic liquid. The mixture was stirred until uniform. Do not over-stir, as this causes the acrylic to set-up prematurely. This mixture was divided into four mounting caps and packed down using a stainless steel spatula.

Another batch of acrylic was prepared using a new cold beaker. One of the four caps previously partially filled, was filled to the top of the cap, packing and leveling the mixture with the spatula. A tooth was then pushed into the center of the cap, while leveling the tooth and the mixture, ensuring a smooth and level surface. With the remaining mixture, another tooth was mounted. When mounting the teeth it is important to keep the tooth surface free of acrylic. This procedure was repeated until the required number of teeth were mounted, to provide approximately 5

samples per cell. The samples were allowed to dry completely (approximately 1 to 2 hours at ambient temperature) and stored in a 100% humidity desiccator, until their use.

C: PREPARATION OF SOAKING SOLUTION

400 mL of solution were prepared by combining deionized water, sodium tripolyphosphate and disodium pyrophosphate or potassium pyrophosphate at the use level required for maximum whitening and mixed continuously.

D: COLORIMETER READING: SOAKING AND DATA ANALYSIS

Mounted teeth were removed from desiccator and allowed to dry at ambient temperature for approximately 45 minutes. After teeth were dry they were scribed with their position number and 4 equal positions to accommodate for variations in the tooth surface. The samples were then read on the colorimeter for pre-soak L values. The data generated were recorded in spread sheet format, using for example, Microsoft Excel®.

After preliminary readings were obtained, the teeth were placed five to a cell, each cell holding samples totaling approximately the same average L value as the next, ensuring that each cell has approximately the same baseline L value. Samples were placed in the mixing solutions in the pre-determined order described above. The samples were allowed to soak for a period of thirty minutes, after which time the samples were removed from the mixing solutions and rinsed thoroughly, to remove any active remaining on the tooth surface.

The samples were allowed to dry at ambient for a period of 45 minutes, after which time the samples were dried and their post-soak 30 minute L values obtained on the colorimeter.

For an experiment that requires multiple runs, simply repeat this procedure. Generally an experiment consists of a thirty minute and a sixty minute run.

Example 7

Efficacy of an STP/DP Combination in an *In-Vitro* Stain Model

The efficacy of a combination of an alkali metal tripolyphosphate (STP)with an alkali metal pyrophosphate, DP (disodium dihydrogen pyrophosphate), was evaluated in an *in vitro* model system which consisted of exposing stained bovine teeth to an aqueous solution of the combination. It will be understood by the skilled artisan that the amount of STP used in this soaking study is 1.25% as a result of the dilution of a 5% STP toothpaste formulation such that the environment of the average oral cavity (containing saliva) is simulated.

The teeth were washed with demonized water and allowed to air dry prior to being mounted into Ecotrin lids using an acrylic polymer, such as jet acrylic powder and jet acrylic liquid from Lang Dental Manufacturing. The teeth were mounted in the center of the lid such that the front of each individual tooth was exposed without there being any resin on the tooth surface. The mounted teeth were stored in a desiccator which had a relative humidity of 100%.

Prior to use, the teeth were removed from the desiccator and allowed to air dry for 45 minutes. The teeth were numbered and then scribed so as to identify, for measurement purposes, ten equal positions on each tooth surface. The presoak L values (average of ten readings per tooth) were determined using a colorimeter (Hunter Labs).

After the presoak readings were taken, the teeth were arranged five per cell, so that teeth having similar L value averages were located in the same cell. This ensured that there was a similar baseline for each solution measured.

The teeth were then stored in test solutions for various time points, typically 30, 60 and 1440 minutes, after which time the teeth were removed, rinsed thoroughly and allowed to air dry for 45 minutes. Test solutions comprising STP (Monsanto Corp.) (1.25% w/w), DP (Albright & Wilson) (2% w/w) and STP/DP (1.25/2% w/w, respectively) were prepared in USP grade water. The pH of the test solutions were adjusted to pH 9 with sodium hydroxide.

The post soak L value averages were then determined as before using a colorimeter and the ΔL values were calculated as indicated below:

$$\Delta L = L_{postsoak} - L_{presoak}$$

The results of the 30 and 60 minute soaking studies, depicted in Fig. 3, show a synergistic effect on the stain removal properties of a combination of STP with DP.

The results of the soaking studies are graphically illustrated in Figs. 1, 1A, 2, 2A and 3. Based upon these results, preferred embodiments of this invention are a composition with about 1.0 to 2.0% disodium pyrophosphate and 5 to 7.5% STP, or a composition with about 3.0 to 4.0% tetrapotassium pyrophosphate and 5 to 7.5% STP. In addition, Fig. 10 supports the conclusion that formulation A is a preferred embodiment for a whitening and stain preventing dentifrice. Each of these particular compositions achieves maximum whitening, above that achieved by a composition with 10% STP alone as the whitening agent.

Example 8

In an adaptation of the prevention of stain build-up *in vitro* perspex block methodology described by Leard A. and Addy M., **1997**, J.Clin. Periodontol *24*, p. 115-118, a stain prevention study using human enamel as substrate (n = 3 per treatment) was conducted.

At the outset of the experiment, the color of the human enamel substrate was measured using a Minolta chromameter. The substrate was then subjected to the following treatment cycle:

Saliva (2 minutes) → DI H_20 Wash → Corsodyl (0.2% chlorhexidine, 2 minutes) → DI H_20 Wash → Test Treatment (2 minutes) → DI H_20 Wash → Tea (1 hour) → DI H_20 Wash. ("DI" is used herein to mean deionized water).

This cycle was repeated 7 times in total; the substrate color was then re-recorded using the Minolta chromameter.

The following test treatments were evaluated for prevention of stain build-up via this methodology:

- Water
- 0.5% PVP
- 1% PVP
- 3% PVP
- 5% PVP
- 10% PVP
- 5% STP
- 10% STP
- 10% PVP/5% STP
- Dentifrice containing 10%STP
- Corsodyl (0.2% Chlorhexidine)
- Dentifrice containing 2% TKPP/0.91% TSPP/5% STP/ 1% PVP (Formula "A")
- Dentifrice containing 3% PVP/5% STP (Formula "B")

A graphical summary of the results obtained from this work are presented in Fig. 9. Changes in color of the human substrate were calculated by subtracting initial chromameter readings from final chromameter data.

A statistical analysis (95% LSD) carried out on the data supports the conclusion that both formulations A and B are numerically and statistically significantly superior at stain prevention versus a dentifrice containing 10%STP alone.

All publications, including, but not limited to, patents and patent applications cited in this specification, are herein incorporated by reference as if each individual publication were specifically and individually indicated to be incorporated by reference herein as though fully set forth.

The above description fully discloses the invention including preferred embodiments thereof. Modifications and improvements of the embodiments specifically disclosed herein are within the scope of the following claims. Without further elaboration it is believed that one skilled in the art can, given the preceding description, utilize the present invention to its fullest extent. Therefore any examples are to be construed as merely illustrative and not a limitation on the scope of the present invention in any way. The embodiments of the invention in which an exclusive property or privilege is claimed are defined as follows.

What is claimed is:

1. A dentally acceptable composition for preventing the build-up, reducing or removing, surface deposited stains from natural teeth and dental prostheses comprising about 0.5% to 10% by weight of a water soluble alkali metal tripolyphosphate salt in combination with about 0.1 and in text to 10% by weight of at least one alkali metal pyrophosphate salt and about 0.1 to 10% by weight of PVP.

2. The composition according to claim 1, wherein the water soluble alkali metal tripolyphosphate is sodium tripolyphosphate.

3. The composition according to claim 2, wherein the sodium tripolyphosphate is present in an amount between about 5 and 7.5% by weight of the total composition.

4. The composition according to claim 1, wherein the alkali metal pyrophosphate salt is selected from tetrasodium pyrophosphate and tetrapotassium pyrophosphate.

5. The composition according to claim 4, wherein the tetrasodium pyrophosphate is present in an amount between about 0.1 and 10% by weight of the total composition.

6. The composition according to claim 4, wherein the tetrapotassium pyrophosphate is present in an amount between about 0.5 and 10% by weight of the total composition.

7. The composition according to claim 1, wherein the PVP is present in an amount between about 0.5 and 5% by weight of the total composition.

8. A method for preventing the build up of, reducing or removing, surface deposited stains from natural teeth and dental prostheses which method comprises contacting teeth or dental prostheses with a dentally acceptable composition comprising about 1 to 10% by weight of a water soluble alkali metal tripolyphosphate salt in combination with about 0.5 to 10% by weight of at least one alkali metal pyrophosphate salt and about 0.1 to 10% by weight of PVP.

9. A method for reducing or removing surface deposited stains from natural teeth and dental prostheses which method comprises contacting teeth or dental prostheses with a dentally acceptable composition comprising about 1 to 10% by weight of a water soluble alkali metal tripolyphosphate salt in combination with about 0.5 to 10% by weight of at least one alkali metal pyrophosphate salt.

10. The method of claim 9, wherein the dentally acceptable composition further comprises about 0.1 to 10% by weight of PVP.

Fig. 1: In vitro Soaking Study
Effect of STP/DSP Combination

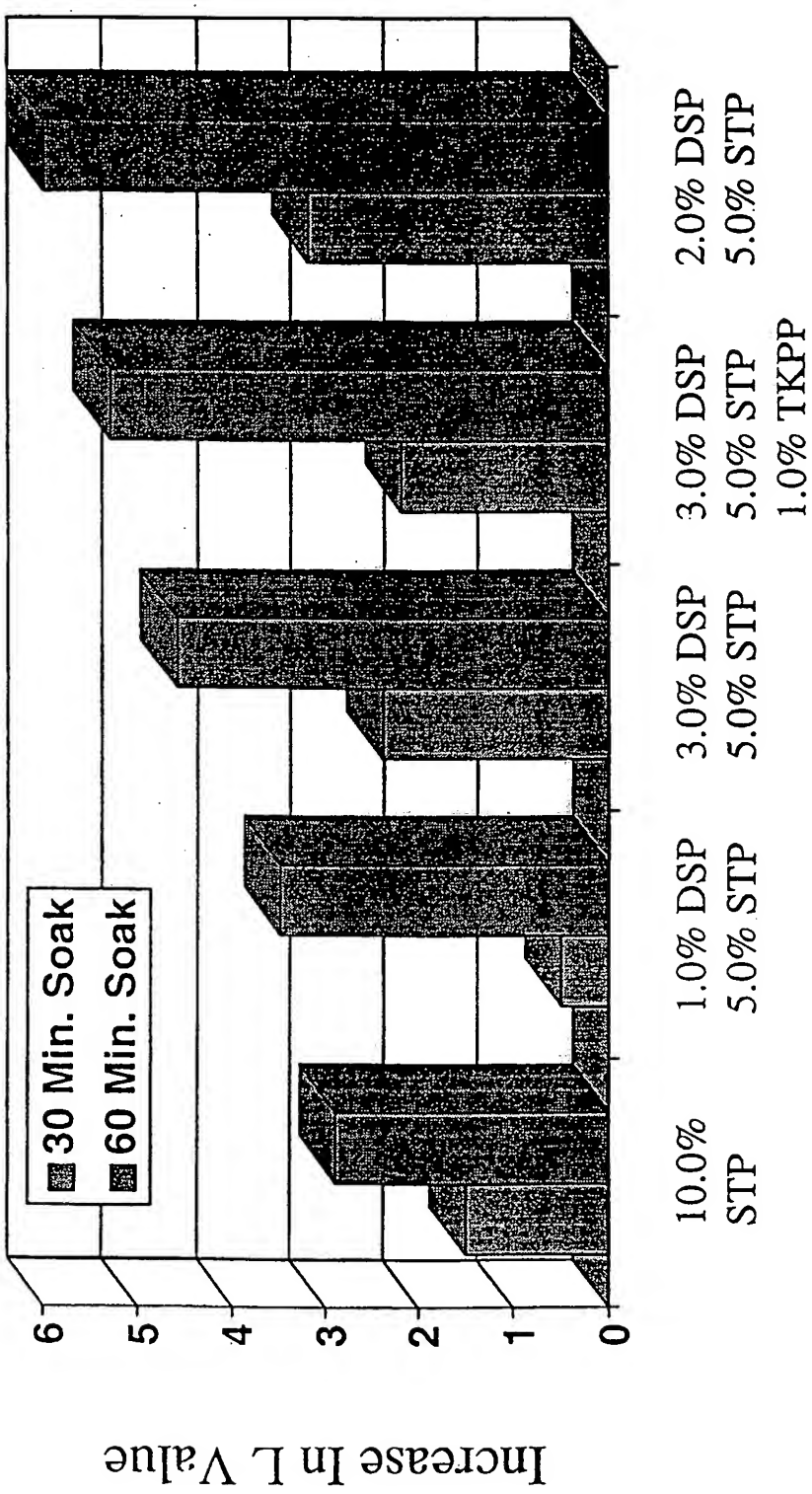
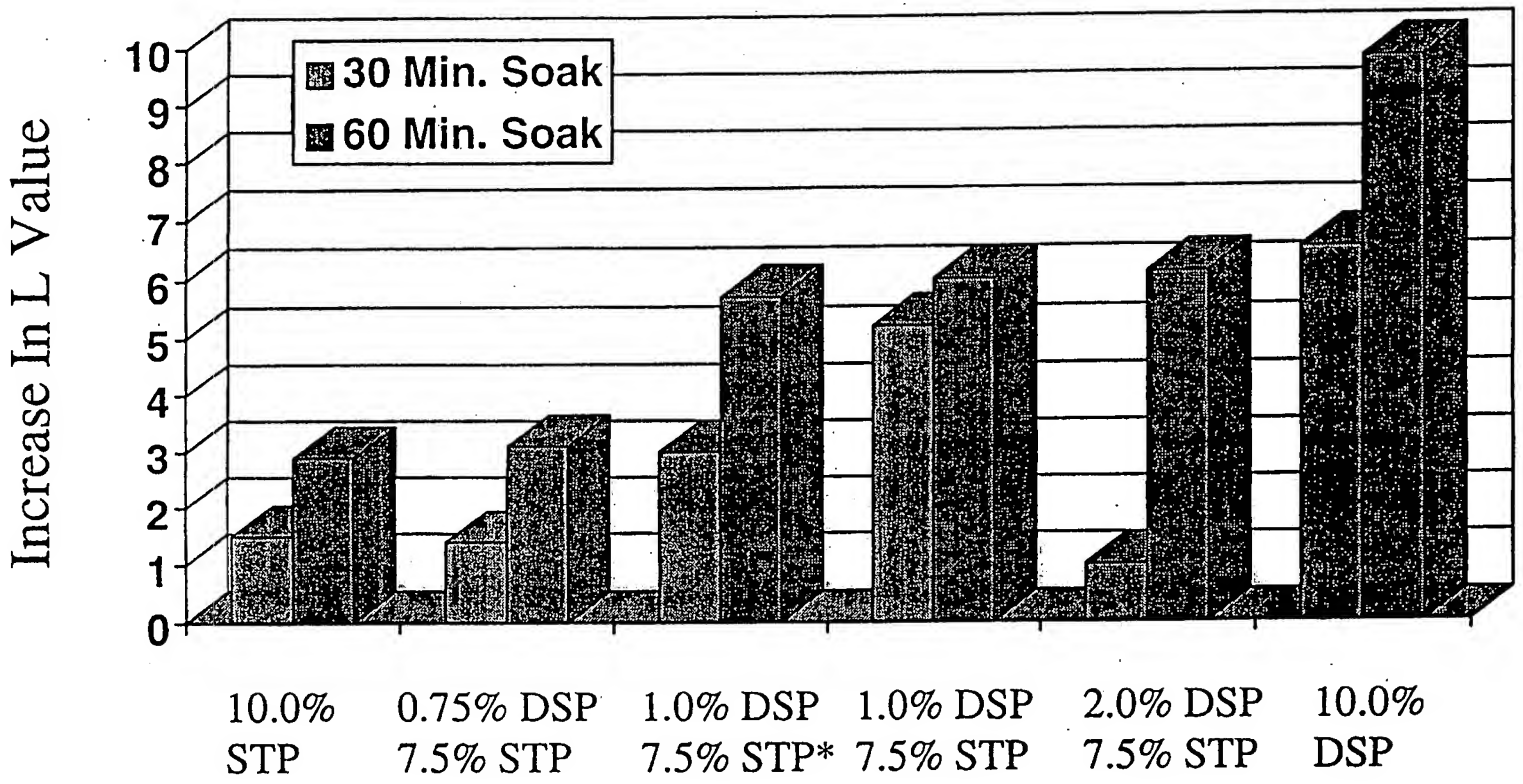
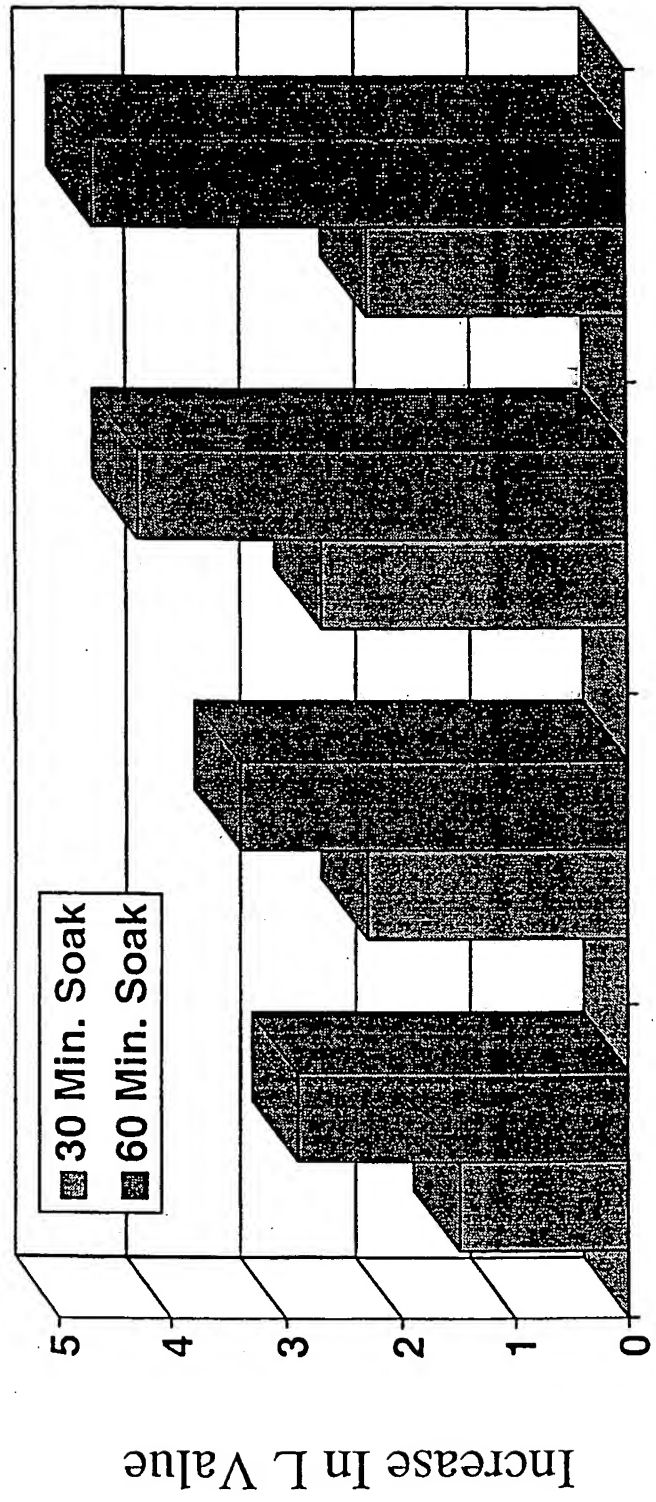


Fig. 1A: In vitro Soaking Study
Effect of STP/DSP Combination



* pH increased w/ NaOl

Fig. 2: In vitro Soaking Study
Effect of STP/TKPP Combination



* With Lactic Acid

Fig. 2A: In vitro Soaking Study
Effect of STP/TKPP Combination

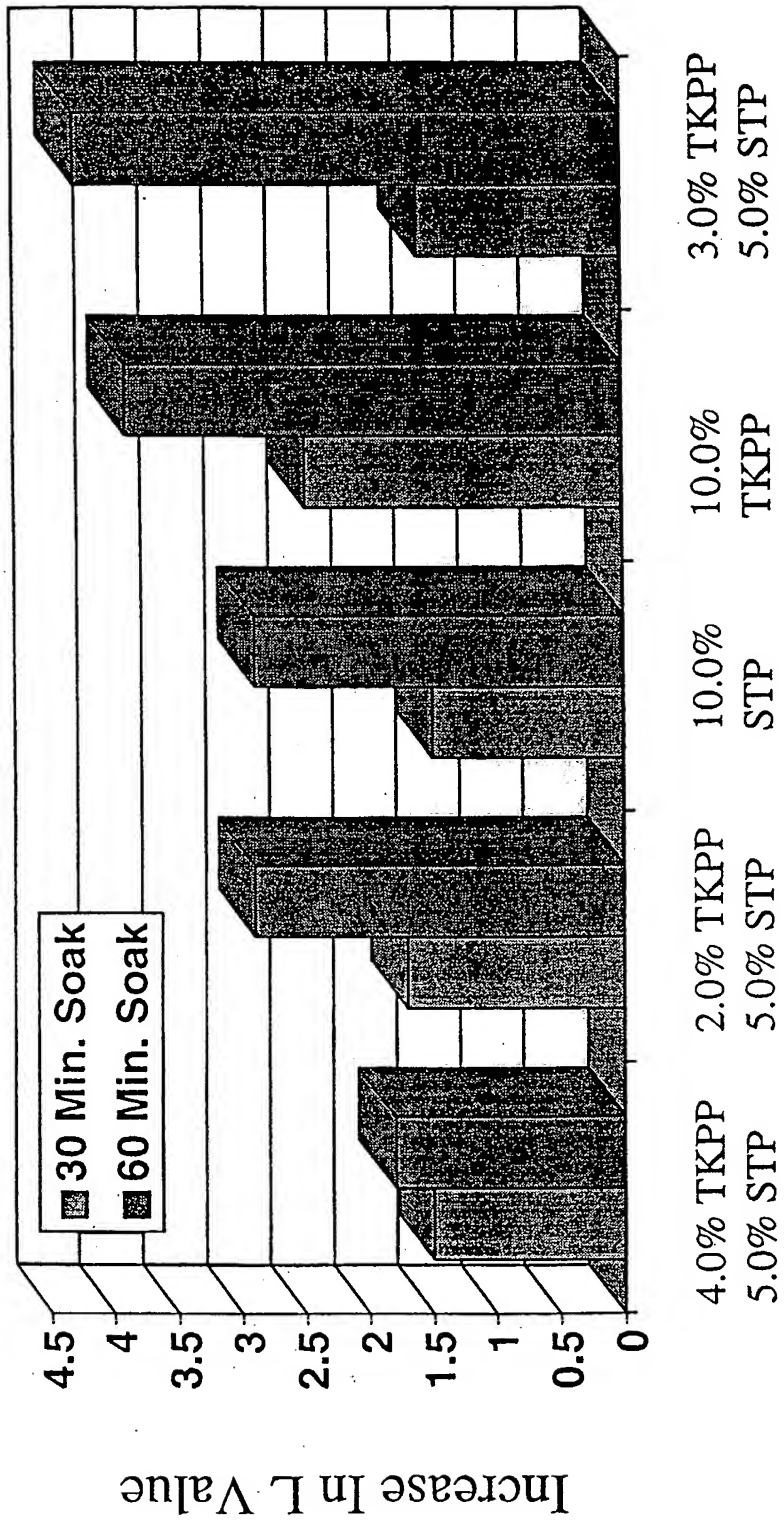
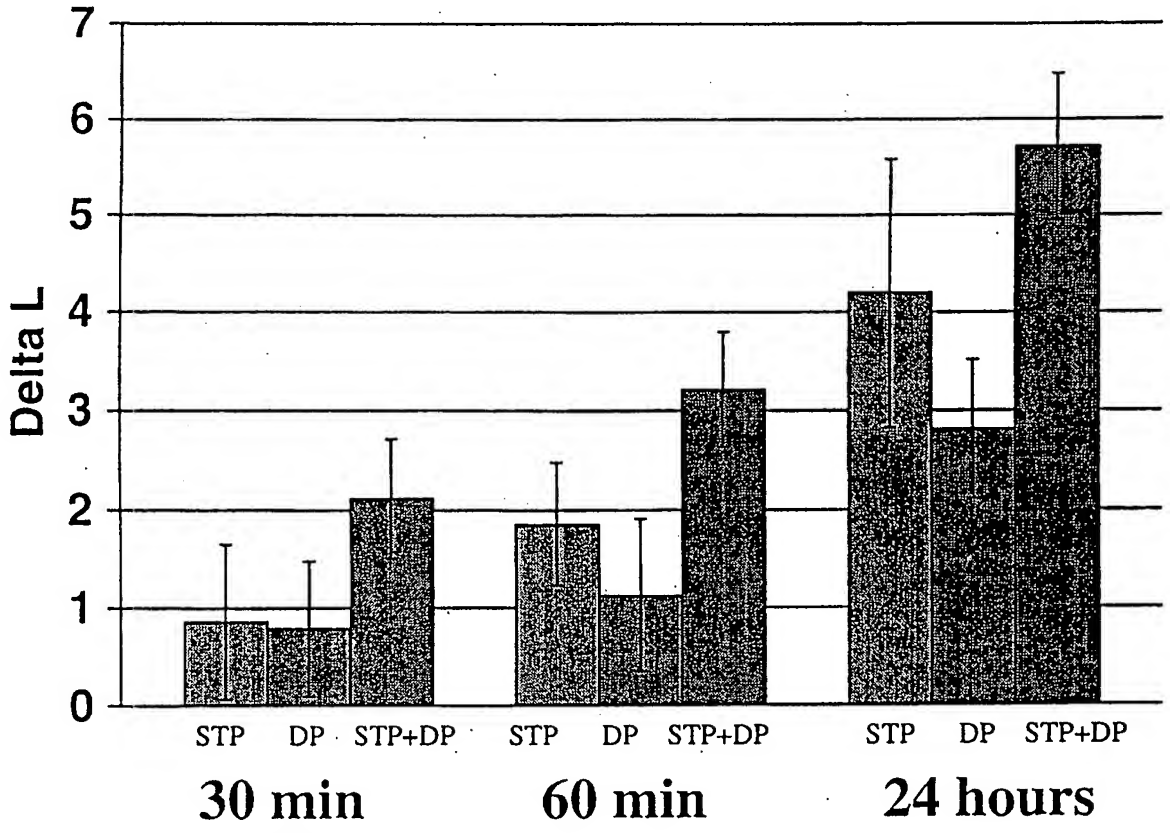


Fig 3: Effect of an STP/DP combination on the removal of stain on bovine enamel (*in-vitro* test)



STP/DP combination 1.25%/2%
STP 1.25%
DP 2%

All solutions approx. pH 9

#1						
Tetra-Sodium Pyrophosphate	Pre Soak	Post Soak 30 Min.	Increase	Post Soak 60 Min.	Increase	Total Increase
pH 10.05 @ 10.0%						
#4	69.49	70.65	1.16	70.96	0.31	1.47
#6	72.91	75.99	3.08	76.22	0.23	3.31
#7	49.46	49.28	-0.18	49.5	0.22	0.04
#11	59.21	63.93	4.72	64.76	0.83	5.55
#13	61.6	61.93	0.33	62.24	0.31	0.64
Average	62.534	64.356	1.822	64.736	0.38	2.202
#2						
Tetra-Potassium Pyrophosphate						
pH 10.23 @ 10.0%						
#1	50.26	50.5	0.24	50.95	0.45	0.69
#3	74.38	75.56	1.18	78.42	2.86	4.04
#5	68.13	69.41	1.28	69.81	0.4	1.68
#14	57.69	65.77	8.08	68.6	2.83	10.91
#15	61.62	63.23	1.61	63.63	0.4	2.01
Average	62.416	64.894	2.478	66.282	1.388	3.866
#3						
Menadione Sodium Bisulphite						
pH 5.77 @ 10.0%						
#2	54.93	55.25	0.32	55.73	0.48	0.8
#8	63.54	63.83	0.29	65.18	1.35	1.64
#9	76.6	76.38	-0.22	75.93	-0.45	-0.67
#10	59.45	60.09	0.64	61.03	0.94	1.58
#12	56.07	56.65	0.58	57.3	0.65	1.23
Average	62.118	62.44	0.322	63.034	0.594	0.916

Fig. 4

#1						
5.0% PVP-/-7.5% STP	Pre Soak	Post Soak 30 Min.	Increase	Post Soak 60 Min.	Increase	Total Increase
pH 8.73						
#3	57.25	61.15	3.9	62.17	1.02	4.92
#5	58.31	59.81	1.5	60.59	0.78	2.28
#12	62.27	67.35	5.08	69.66	2.31	7.39
#13	50.35	51.21	0.86	51.41	0.2	1.06
#15	48.77	49.94	1.17	50.61	0.67	1.84
Average	55.39	57.892	2.502	58.888	0.996	3.498
#2						
10.0% DiSodium Pyrophosphate						
pH 3.76						
#1	61.51	65.68	4.17	68.8	3.12	7.29
#2	55.97	61.78	5.81	63.96	2.18	7.99
#6	59.08	63.42	4.34	66.39	2.97	7.31
#7	56.93	68.78	11.85	72.12	3.34	15.19
#8	42.82	49.3	6.48	53.87	4.57	11.05
Average	55.262	61.792	6.53	65.028	3.236	9.766
#3						
3.0% TKPP-/-7.5% STP						
pH 8.15						
#4	69.48	73.24	3.76	74.36	1.12	4.88
#9	52.54	54.59	2.05	56	1.41	3.46
#10	57.92	61.23	3.31	65.65	4.42	7.73
#11	46.49	48.3	1.81	48.76	0.46	2.27
#14	57.27	59.8	2.53	60.53	0.73	3.26
Average	56.74	59.432	2.692	61.06	1.628	4.32

Fig. 5

#1						
3.0%TKPP/7.5%STP/Lactic Acid	Pre Soak	Post Soak 30 Min.	Increase	Post Soak 60 Min.	Increase	Total Increase
pH 7.9						
#1	70.73	73.04	2.31	73.64	0.6	2.91
#5	60.62	60.95	0.33	61.63	0.68	1.01
#7	49.3	51.37	2.07	51.76	0.39	2.46
#9	56.74	57.72	0.98	58.81	1.09	2.07
#14	65.23	70.92	5.69	74.02	3.1	8.79
Average	60.524	62.8	2.276	63.972	1.172	3.448
#2						
4.0% TKPP-/-5.0% STP						
pH 9.4						
#2	54.89	56.51	1.62	57.5	0.99	2.61
#3	64.98	66.94	1.96	66.61	-0.33	1.63
#10	59.56	60.82	1.26	61.02	0.2	1.46
#13	51.19	52.17	0.98	52.96	0.79	1.77
#16	70.59	72.12	1.53	72.1	-0.02	1.51
Average	60.242	61.712	1.47	62.038	0.326	1.796
#3						
1.0% Di Na Pyro-/-7.5% STP						
pH 7.5						
#11	48.84	50.12	1.28	50.56	0.44	1.72
#12	56.81	58.51	1.7	58.97	0.46	2.16
#17	59.98	66.6	6.62	67.61	1.01	7.63
#18	63.82	66.46	2.64	67.31	0.85	3.49
#19	70.66	84.51	13.85	85.86	1.35	15.2
Average	60.022	65.24	5.218	66.062	0.822	6.04
#4						
Colgate Whitening						
pH 8.8						
#8	53.21	53.07	-0.14	54.37	1.3	1.16
#15	50.71	50.88	0.17	50.99	0.11	0.28
#21	66.91	67.64	0.73	67.99	0.35	1.08
#23	58.55	59.7	1.15	60.97	1.27	2.42
#24	71.22	74.47	3.25	73.83	-0.64	2.61
Average	60.12	61.152	1.032	61.63	0.478	1.51

Fig. 6

#1						
.75% Di Na Pyro.-/-7.5% STP	Pre Soak	Post Soak 30 Min.	Increase	Post Soak 60 Min.	Increase	Total Increase
pH 7.9						
#1	73.64	74.49	0.85	74.68	0.19	1.04
#3	66.61	68.05	1.44	69.98	1.93	3.37
#5	61.63	63.01	1.38	65	1.99	3.37
#7	51.76	53.8	2.04	53.88	0.08	2.12
#9	58.81	60.2	1.39	64.56	4.36	5.75
Average	62.49	63.91	1.42	65.62	1.71	3.13
#2						
1.0% Di Na Pyro-/-7.5% STP						
pH 8.08 Increased w/ NaOH 0.45%						
#2	57.5	58.57	1.07	61.48	2.91	3.98
#10	61.02	62.21	1.19	63.48	1.27	2.46
#11	50.56	51.92	1.36	55.36	3.44	4.8
#17	67.61	70.33	2.72	74.92	4.59	7.31
#24	73.83	82.45	8.62	83.54	1.09	9.71
Average	62.104	65.096	2.992	67.756	2.66	5.652
#3						
4.0% TKPP-/-7.5% STP						
pH 9.31						
#12	58.97	60.43	1.46	62.58	2.15	3.61
#15	50.99	52.69	1.7	53.01	0.32	2.02
#16	72.1	74.12	2.02	74.54	0.42	2.44
#18	67.31	69.14	1.83	69.93	0.79	2.62
#23	60.97	65.36	4.39	73.72	8.36	12.75
Average	62.068	64.348	2.28	66.756	2.408	4.688

Fig. 7

#1	Pre Soak	Post Soak 30 Min.	Increase	Post Soak 60 Min.	Increase	Total Increase
3.0% DiNa Pyro-/-5.0% STP						
pH 8.19 increased w/ 1.8% NaOH						
#2	67.74	68.28	0.54	70.83	2.55	3.09
#3	66.78	68.05	1.27	70.35	2.3	3.57
#5	71.83	72.54	0.71	74.66	2.12	2.83
#6	72.09	73.49	1.4	75.45	1.96	3.36
#17	72.89	81.12	8.23	83.23	2.11	10.34
Average	70.266	72.696	2.43	74.904	2.208	4.638
#2						
3.0% DiNa Pyro-/-1.0% TKPP-/-						
5.0% STP						
pH 8.26 increased w/ 1.8% NaOH						
#1	75.87	78.19	2.32	82.38	4.19	6.51
#4	73.71	73.78	0.07	76.78	3	3.07
#10	63.03	65.74	2.71	67.05	1.31	4.02
#13	74.14	76.98	2.84	80.74	3.76	6.6
#15	69.48	72.43	2.95	75.71	3.28	6.23
Average	71.246	73.424	2.178	76.532	3.108	5.286
#3						
2.0% DiNa Pyro-/-5.0% STP						
pH 8.14 increased w/ 1.10% NaOH						
#7	78.31	81.52	3.21	84.35	2.83	6.04
#8	68.72	73.48	4.76	76.92	3.44	8.2
#9	73.85	76.45	2.6	79.95	3.5	6.1
#11	70.09	71.7	1.61	72.88	1.18	2.79
#12	62.78	66.82	4.04	69.76	2.94	6.98
Average	70.75	73.994	3.244	76.772	2.778	6.022
#4						
3.0% TKPP-/-5.0% STP						
pH 9.38						
#14	68.59	69.41	0.82	71.28	1.87	2.69
#16	71.5	72.33	0.83	75.19	2.86	3.69
#18	64.93	67.79	2.86	70.47	2.68	5.54
#19	76.1	78.01	1.91	81.64	3.63	5.54
#20	72.23	73.8	1.57	76.41	2.61	4.18
Average	70.67	72.268	1.598	74.998	2.73	4.328

Fig. 8

Fig. 9

GRAPH TO SHOW STAIN PREVENTATIVE PROPERTIES OF VARIOUS TOOTHPASTES & SOLUTIONS

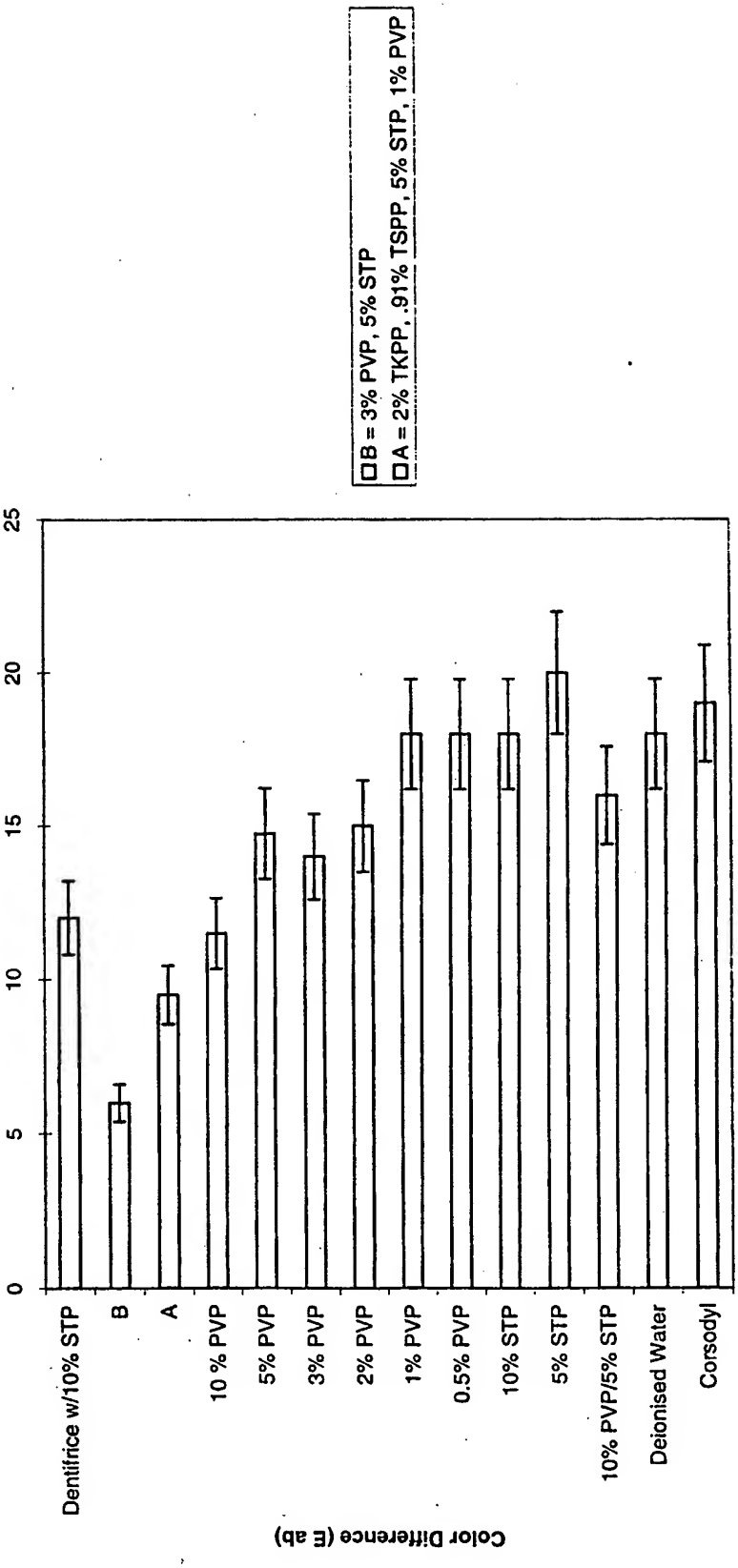
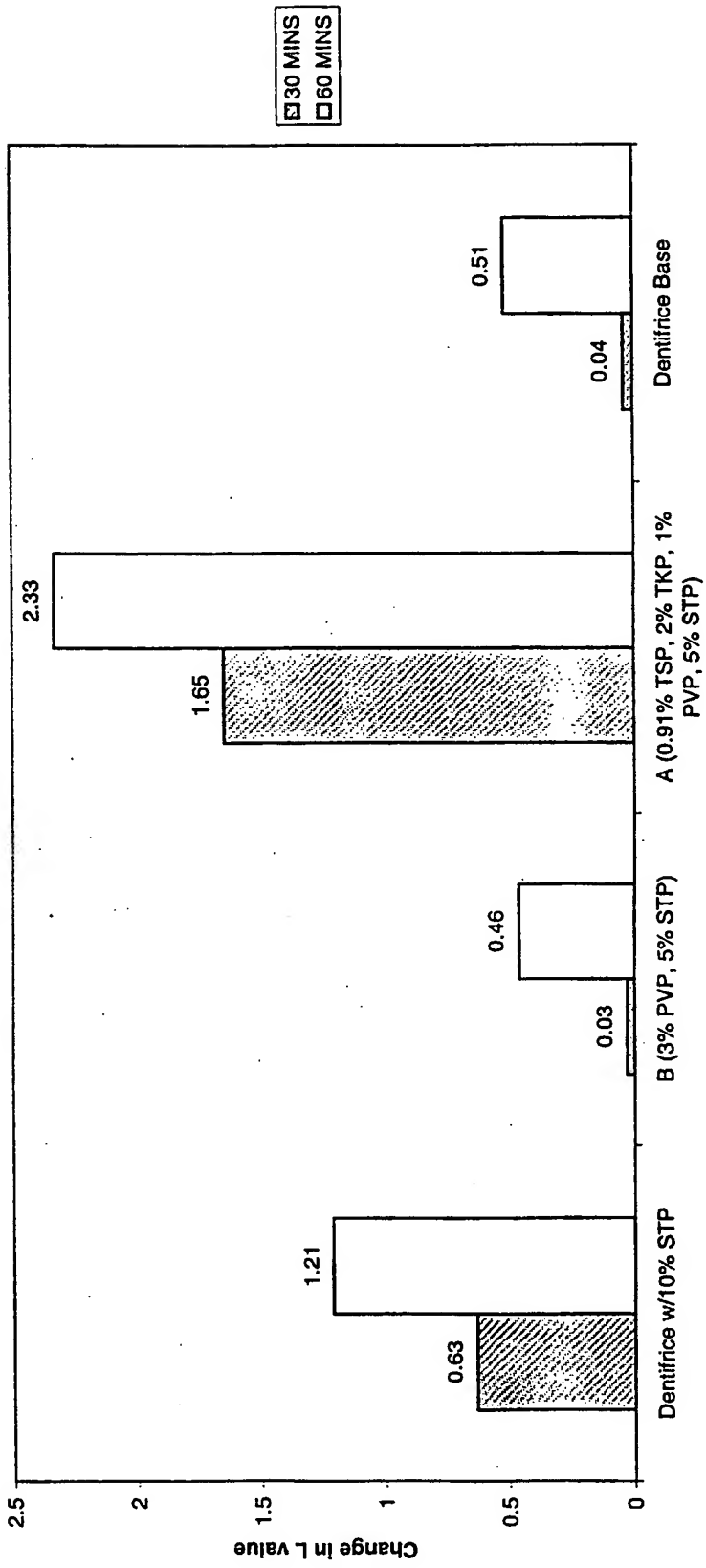


Fig. 10


SOAKING STUDY PERFORMED ON VARIOUS WHITENING TOOTHPASTES
(30 MINS & 60 MINS)
30 MINS
60 MINS
Change In L value
2.5
2
1.5
1
0.5
0
0.63
1.21
0.03
0.46
1.65
2.33
0.04
0.51
Dentifrice w/10% STP
B (3% PVP, 5% STP)
A (0.91% TSP, 2% TKP, 1% PVP, 5% STP)
Dentifrice Base

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US98/18309

A. CLASSIFICATION OF SUBJECT MATTER
IPC(6) :A61K 7/16
US CL :424/49, 57
According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)
U.S. : 424/49, 57

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
Derwent WPIDS

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X -- Y	US 5,176,901 A (GALLOPO et al.) 05 January 1993 (0.1%-15% PVP, 5%-10% STP, TSPP, TKPP), see entire document.	1-7, 8-10
T,P	WO 97/45096 A1 (SMITHKLINE BEECHAM CORPORATION) 04 December 1997, (1%-30% PVP to counter tooth staining, pH 5 to 9).	1-10
Y	WO 95/05148 A1 (SMITHKLINE BEECHAM CORPORATION) 23 February 1995 (1%-20% alkal metal salts of pyrophosphates anchor tripolyphosphates, e.g. STP, to whiten teeth, pH range 8 to 10), see entire document.	1-10
Y	WO 95/17158 A1 (SMITHKLINE BEECHAM CORPORATION) 29 June 1995 (0.1%-5.5% PVP and 1% to 20% STP or other water soluble alkali metal tripolyphosphate, to whiten teeth), see entire document.	1-10

[X] Further documents are listed in the continuation of Box C. [ ] See patent family annex.

*	Special categories of cited documents:	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
"A"	document defining the general state of the art which is not considered to be of particular relevance	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"E"	earlier document published on or after the international filing date	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"&"	document member of the same patent family
"O"	document referring to an oral disclosure, use, exhibition or other means		
"P"	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search	Date of mailing of the international search report
07 DECEMBER 1998	21 JAN 1999
Name and mailing address of the ISA/US Commissioner of Patents and Trademarks Box PCT Washington, D.C. 20231 Facsimile No. (703) 305-3230	Authorized officer SHEP ROSE aco Telephone No. (703) 308-1235

Form PCT/ISA/210 (second sheet)(July 1992)*

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US98/18309

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	WO 93/16681 A (SMITHKLINE BEECHAM P.L.C.) 02 September 1993 (1% to 30% PVP to counter tooth staining), see entire document.	1-10
Y	GB 741,315 A (COLGATE-PALMOLIVE) 30 November 1955 (1% to 50% PVP to eliminate tooth stains), see entire document.	1-10
Y	US 4,822,599 A (MITRA, S.) 18 April 1989 (1% to 6% TKPP and/or TSPP soluble pyrophosphate salts reduce natural stains on teeth), see entire document.	1-10
A	WO 93/19728 A (SMITHKLINE BEECHAM CORPORATION) 14 October 1993 (DENTAL FORMULATION: 5% STP, 1.8% TSPP and 4.0% TKPP).	1-10
A	EP 0 626 843 B1 (SMITHKLINE BEECHAM PLC) 12 July 1994 (1% to 30% PVP has been proposed to inhibit the formation of and to remove stains from teeth in G.B. 741315 and GB 739936).	1-10
A	US 5,145,667 A (IBRAHIM et al.) 08 September 1992 (4% to 6% STP, pH 8 to 9).	1-10
A	US 4,985,236 A (IBRAHIM et al.) 15 January 1991 (4% to 6% STP, pH 8 to 9).	1-10
A	US 4,923,684 A (IBRAHIM et al.) 08 May 1990 (4% to 6% STP, pH 8 to 10).	1-10
A	EP 0 397 452 A2 (BEECHAM INC.) 14 November 1990 (4% to 6% STP, pH 8 to 10).	1-10